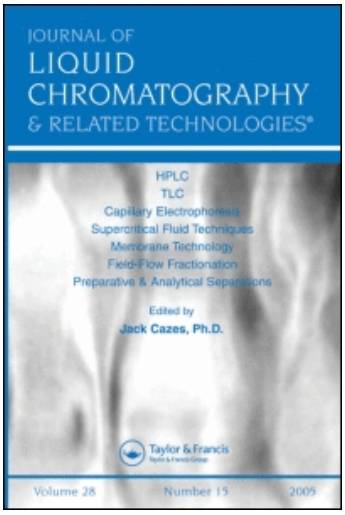

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Analysis of Ginkolides and Biobalides by Capillary Electrophoresis

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ANALYSIS OF GINKOLIDES AND BIOBALIDES BY CAPILLARY ELECTROPHORESIS

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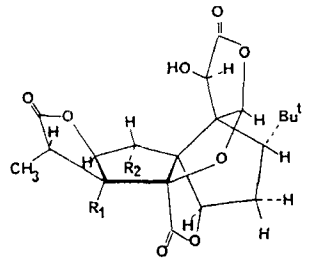
ABSTRACT

A method has been developed to analyze for biobalide, ginkolide A and ginkolide B by capillary electrophoresis (CE). Analysis was accomplished by using a phosphate and sodium dodecyl sulfate (SDS) buffer with direct UV detection at 185nm. Run times of less than eighteen minutes for the compounds of interest was possible.

INTRODUCTION

Interest in analyzing for ginkolides is due to the fact that they possess unique pharmacological properties.[1] Ginkolides occur naturally in leaves of the tree *Ginko biloba* and up until now have been analyzed for exclusively by HPLC with either UV or refractive index (RI) detection.[1-3] Capillary electrophoresis (CE) was investigated as a possible alternative or confirmatory method for these compounds. Figure 1 shows the structures of the compounds under investigation. The purpose of this paper is to report on a preliminary method that has been developed to analyze for these compounds.

GINKOLIDES



	R_1	R_2
Ginkolide A	OH	H
Ginkolide B	OH	OH

BIOBALIDE

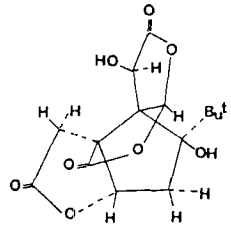


Figure 1. Structure of terpenes from *Ginko biloba* that were investigated.

EXPERIMENTAL

Chemicals

Standards of biobalide, ginkolide A, and ginkolide B were obtained from Sigma (St. Louis, MO) and were used as is. A mix of various terpenes was provided by a producer of terpene mixes and used as received. Phosphate and sodium dodecyl sulfate (SDS) was obtained from Waters Corporation (Milford, MA). High purity water was obtained from a Milli-Q water system (Millipore, Bedford, MA). Methanol used in dissolving the samples and standards was of HPLC grade.

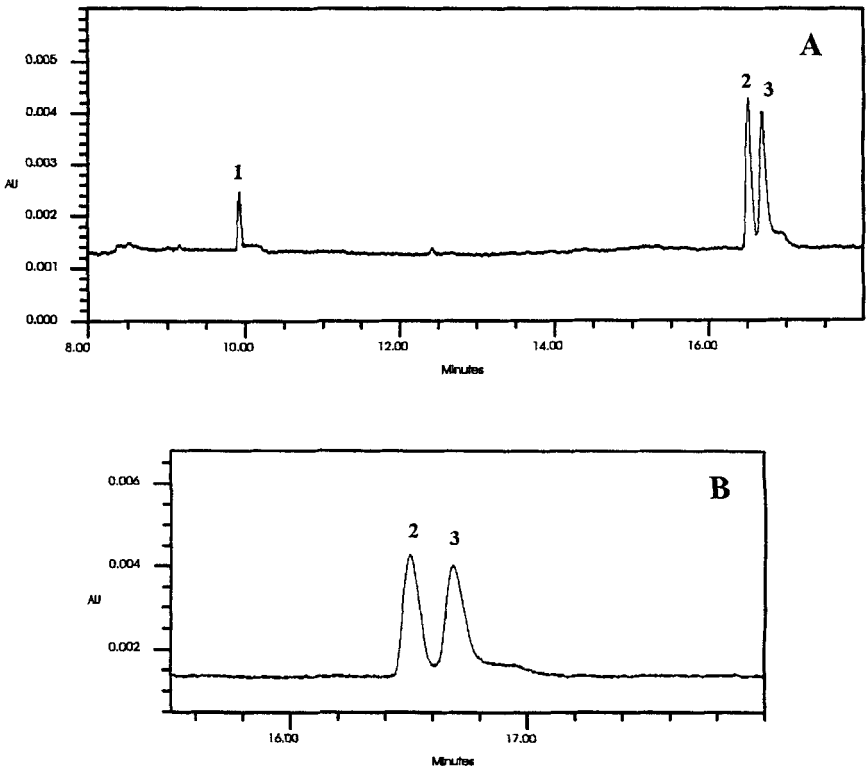


Figure 2. Electropherogram of terpene standards (A) with a close-up of ginkolide A and B separation shown in figure 2B. Peaks: **1**: biobalide; **2**: ginkolide A; **3**: ginkolide B. Amounts of each are 0.12 mg/mL. Conditions as stated in text.

Buffers and Solutions

A standards solution (1.0 mg/mL) of all ginkolides and biobalide was prepared by dissolving them in methanol. Prior to CE analysis the standard solution was diluted in the running buffer resulting in a mix with a concentration of approximately 0.12 mg/mL of each compound. The terpene mix was prepared in a similar manner with a final concentration of the mix being 0.11 mg/mL. The buffer used for the final CE analysis was a mixture of phosphate and SDS at a concentration of 25 mM and 90 mM respectively.

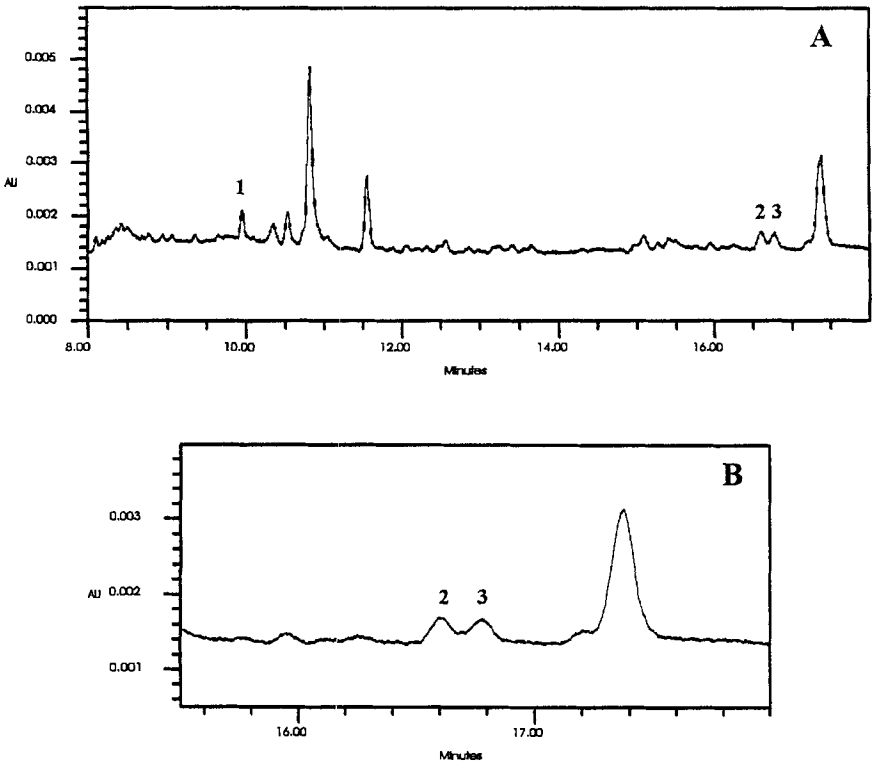


Figure 3. Electropherogram of terpene mix at a concentration of 0.11 mg/mL (A) with a close-up of ginkolide A and B separation shown in figure 3B. Peaks: **1**: biobalide; **2**: ginkolide A; **3**: ginkolide B. Conditions as stated in text.

Instrumentation

The capillary electrophoresis (CE) system used was a Quanta 4000e (Waters Corporation, Milford, MA) with a positive power supply. A Hg lamp was used for direct UV detection at 185 nm. Accusep polyimide fused-silica capillaries of dimension 60 cm X 75 um I.D. were used throughout and obtained from Waters.

Data acquisition and control of the CE was carried out with a Waters Millennium 2010 chromatography manager. The detector time constant was set at 0.3 seconds and the data collection rate was 5 points/sec. The temperature inside the CE was maintained at 30 C.

RESULTS

Due to the compounds low absorbance, 185 nm was chosen as the wavelength to monitor. Initial work using a borate, boric acid, and SDS buffer proved unsuccessful for separating these compounds. A phosphate, and SDS buffer was investigated with a reasonable separation accomplished at 25 mM phosphate and 90 mM SDS. Figure 2 is an electropherogram of the standard mix. Due to the high concentration of components a sampling time of only 5 seconds was used. Figure 2A is a close-up of the ginkolide A and B separation showing good resolution between them. Using this method a terpene sample was analyzed. Figure 3 is an electropherogram of the separation with the compounds of interest identified. In this case the sampling time was raised to 15 seconds.

CONCLUSIONS

As shown in the previous examples, analysis for biobalide, ginkolide A and ginkolide B can be done by CE. Further, CE offers an alternative method for analyzing these compounds with relatively fast run times and rood resolution.

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